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Ice Lithography for Nanodevices

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Abstract. Water vapor is condensed onto a cold sample, coating it with a thin-film of ice. The ice is sensitive to electron beam lithography exposure. 10 nm ice patterns are transferred into metals by “melt-off”. Non-planar samples are coated with ice, and we pattern on cantilevers, AFM tips, and suspended nanotubes.

The Harvard Nanopore Group is one of the pioneers in nanopore DNA sequencing [1]. For solid-state nanopore DNA sequencing, we needed to fabricate a chip with a carbon nanotube field effect transistor traversing a nanopore. Originating from electron beam lithography PMMA resist, organic contamination a few nanometers thick, also called scum, partially bury nanotubes and clogs the nanopore just a few nanometers in diameter. While oxygen plasma is effective in removing scum, it also destroys the nanotubes. We discovered that it is possible to pattern ice with electron beam lithography (EBL), and named the method “ice lithography” [2]. Here, the main advantage is that water does not contaminate like organic resists.

Fig. 1 illustrates ice lithography for patterning the electrodes of a nanotube field effect transistor. We modified a scanning electron microscope (SEM) for dedicated ice lithography [3]. In brief, we added a load lock for sample loading into the SEM vacuum chamber; a gas injection system to inject water vapor into the SEM vacuum chamber; a cryogenic system to cool the sample and condense water vapor from the GIS; a pattern generation system to guide the electron beam for writing in ice; and a metal deposition system for sputtering metals onto samples with ice patterns. After metal coating, the sample is unloaded from the instrument and immersed into isopropanol for “melt-off”. The ice melts, metal covering the ice is removed from the sample, while metal in patterned areas with no ice remains.

Unlike uniformly spinning resists that require a planar substrate, we can condense water vapor on non-planar surfaces and fragile samples and subsequently pattern [4]. We took advantage of this unique property to pattern on silicon nitride and oxide membranes down to 20-nm-thin. Because thin membranes are transparent to 200 keV electrons, it is possible to image metal patterns

made by ice lithography by transmission electron microscopy (TEM). The smallest patterns are less than 10 nm (Fig 2a). The line edge roughness is so small that it is hardly measurable in the TEM images.

Using the same approach, we patterned on cantilevers and the apex of atomic force microscope probes tips. Such samples do not allow resist spin and bake.

To further test ice lithography for fragile and non-planar samples, we patterned on suspended single walled carbon nanotube 5 nm in diameter and 1000 nm long. We coated the nanotube with 20 nm of ice film, then segments of ice along the nanotube were removed. Each segment was 25 nm wide, and the distance between segments was 150 nm. 5 Å of Ti was deposited over the entire sample, and by melt-off we made segment of Ti. Upon exposure to air, Ti forms TiO₂, which seeds the growth of alumina by atomic layer deposition. We formed pearls (alumina particles) on a string (nanotube) structure (Fig 2b).

We also made metal contacts to carbon nanotube for field effect transistors, with resulting electrical properties similar to contacts made by organic EBL resists [5].

We showed that ice lithography could be uniquely applied to make pristine carbon nanotube field effect transistors, and pattern on fragile samples with non-planar geometries, and hence enables the construction of a new generation of nanostructures with versatile functionalities. Ice lithography, however, has some drawbacks: the exposure dose is 1000 times higher than PMMA. For our work, less than 30 minutes is dedicated to ice resist exposure, and we save time because all processes are done using the ice lithography instrument. While it is clearly not yet suitable for production of semiconductor devices, we believe ice lithography has unique advantages in a research perspective. At the

National Micro and Nanofabrication facility DTU Danchip we are setting up an new ice lithography instrument, and at the DTU core electron microscopy facility DTU Cen, we will study electron ice interactions, which is an exciting and unexplored area.

Acknowledgements

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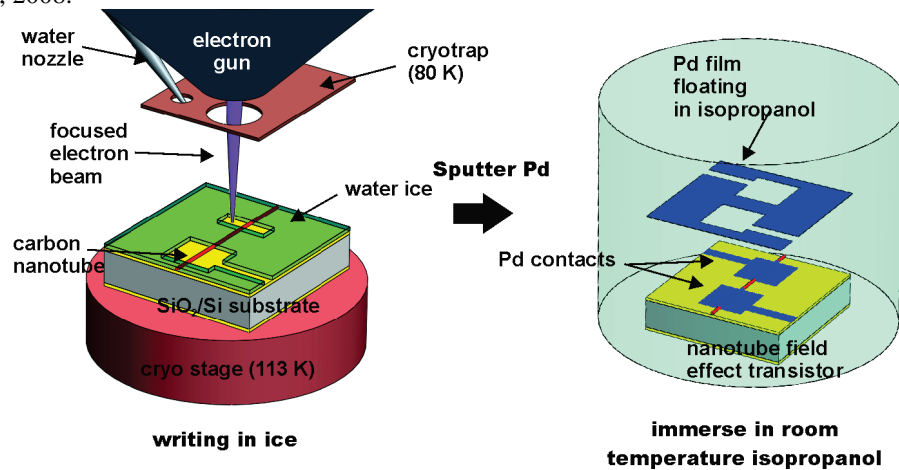


Figure 1. Ice lithography process illustration

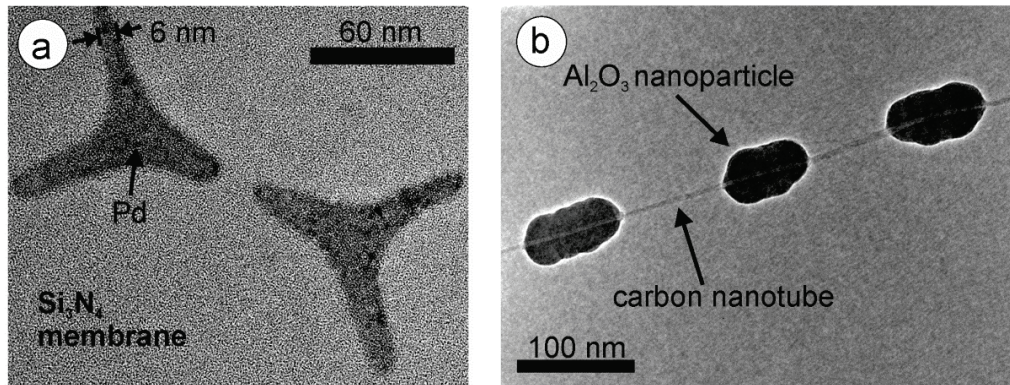


Figure 2. (a) TEM image of Pd structures on 50-nm-thin silicon nitride film, and nanoparticles on nanotubes (b).



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